

Application No. 09/846,654

CASE 8539

## IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Applicant : **Jonathan Richard Clare, et al.**  
Application Serial No. : **09/846,654**  
Confirmation No. : **4546**  
Filed : **May 1, 2001**

Title: **AUTOMATIC DISHWASHING COMPOSITIONS COMPRISING DIACYL  
PEROXIDE BLEACH AND BLOOMING PERFUME**

Group Art Unit : **1751**  
Examiner : **John R. Hardee**  
Docket No. : **8539/klw**

DECLARATION UNDER 37 CFR 1.132

I, Brian X. Song, do hereby declare the following:

1. That I received a B.S. Degree in Chemistry from Peking University in 1983, an M.S. Degree in Chemistry from Peking University in 1986, and a Ph.D. Degree in Chemistry from the Academia Sinica in Beijing, China in 1989. Afterwards, I carried out a post-doctoral appointment with Wayne State University in Detroit from 1989-93, and a post-doctoral appointment with the University of Illinois at Urbana-Champaign from 1993-1995. Since 1995, I have worked for The Procter & Gamble Company in the fields of detergents and dish care.

2. I further declare that the following measurement was carried out at my direction. A diacyl peroxide particle size distribution for benzoyl peroxide (BPO), Oxycare 50 (ultra micronized), as measured by Horiba LA-910 Laser Scattering Particle Size Distribution Analyzer show that the particle size distribution falls within the Applicants' claimed range of from about 0.1 to less than 10 microns (see Appendix I). In this graph, the x-axis is particle size measured in microns ( $\mu\text{m}$ ), the y-axis is the cumulative % undersize. The particle size distribution shows that about 99% of the particles are equal or less than 1 micron (number average distribution). Appendix II is a graph of the volume average (or volume mode) particle size distribution of an ultra micronized benzoyl peroxide material revealed in the present application. This data shows this material has a mean particle size of 4.1 microns. In addition, the graph shows that more than 90% of all the ultra micronized benzoyl peroxide material described in the present application is below 10

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microns. This is in contrast to the preferred particle size of 10 to 100 microns disclosed by in the Painter et al. patents, U.S. Patent Number 5,763,378, and U.S. Patent Number 5,902,781. Furthermore, '378 disclose specific examples of a method for cleaning plasticware comprising diacyl peroxide particle flakes having a particle size of 53, 65, 67 and 68 microns. (See, Painter '378, Table I, col. 26, lines 30-36). Net, even though the references revealed a broad particle size range of from 1 to 300 microns, it is quite clear that the referenced patents were not in possession of our invention, that is, diacyl peroxide particles below 10 microns.

3. I further declare that on December 20, 2001 the following experiment was carried out by employees of the Procter & Gamble Company in order to demonstrate the effect of particle size of diacyl peroxide on soiled plasticware, in the presence of regular automatic dishwashing detergent. Rubbermaid spatulas were stained with tomato sauce (Spaghetti sauce) by placing them in heated beaker at 185 degrees F for 30 seconds. The spatulas were taken out and allowed to cool and dry. Color intensities of the stained spatulas were recorded by an image analysis system comprised of a Dell computer, Sony 3CCD Camera and Sony DX C-755 Camera Control, a specially designed lighting system, and Optimas Software version 6.21.19 from Media Cybernetics, L.P. The stained spatulas were placed in the cutlery basket of the GE500. The open dispenser cup was then filled with about 40 grams of the composition. The closable dispenser cup was filled with regular automatic dishwashing detergent (eg Cascade powder). A normal cycle with heated dry was run. At the end of the wash, the spatulas were removed from the automatic dishwashing machine and reanalyzed for color intensity by image analysis. Percent stain removal was then calculated based on the color intensity change of the spatulas before and after the wash. See Table 1 for the stain removal results.

Table 1. (Test 12/20/2001) Formulation according to the invention containing different Oxycare raw materials (treatment A and B)

Treatment	Mean particle size, microns* (Volume mode)	% stain removal on spatulas
A. Ultra-micronized BPO (Oxycare 42, lot 38837)	4.2	82 B**
B. Regular micronized BPO (lot 37199)	22.2	59
LSD (95%)	--	10

\*Particle sizes were obtained by Horiba LA910 particle size analyzer. Stain removal testing was done in four GE 500 machines, 4 cycles with rotation (i.e., each treatment sees each machine once). Each machine contains two spatulas, both sides of the spatulas were imaged. LSD is calculated using a SAS program.

B\*\* means treatment A is significantly better than treatment B.

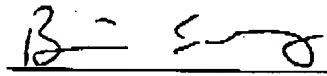
The first composition tested consisted of an ultra micronized BPO material (@ 3.6% active) with a mean particle size of 4.2 microns. The particle size distribution (volume mode) of the BPO material is shown in Appendix II which shows about 90% of the BPO material is below 10 microns. The second composition tested consisted of a BPO material (also @ 3.6% active) with a mean particle size of about 22 microns. Clearly, these results showed that the first composition consisting of a BPO material with mean particle size of 4.2 microns (less than 10 microns) removes the stains significantly better (@95% confidence) than the second composition consisting of a BPO material with a mean particle size of 22.2 microns (greater than 10 microns).

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4. Further, I declare that I reviewed the September 27, 1999 experiment carried on by Marie Salem on the effect of diacyl peroxide particle size on stain removal performance and film grade on plasticware (in the absence of regular automatic dishwashing detergent), it is my expert opinion that aqueous wash liquor in automatic dishwashing machines containing diacyl peroxide particles having a particle size of from about 0.1 to less than 10 microns provide significantly better stain removal performance on stained plasticware and less film formation on clear plasticware as compared to aqueous wash liquor in automatic dishwashing machines containing diacyl peroxide particles having a mean particle size of at least 10 microns. It is further my opinion that one of ordinary skill in the art in reviewing Painter et al., U.S. Patent Number 5,763,378, as well as, Painter et al., U.S. Patent Number 5,902,781, would not expect to achieve this significantly improved stain removal performance and reduction in film formation on plasticware, and therefore, would not be motivated to utilize diacyl peroxide particles having a particle size of from about 0.1 to less than 10 microns.

The undersigned declares that all statements made herein which are of declarant's own knowledge are true and that all statements made on information and belief are believed to be true; that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

Further declarant sayeth not.

  
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Brian X. Song

Date: 8/16/03

18 USC 1001. Whoever, in any matter within the jurisdiction of any department or agency of the United States knowingly and willfully falsifies, conceals or covers up by any trick, scheme, or device a material fact, or makes any false, fictitious or fraudulent statements or representations, or makes or uses any false writing or document knowing the same to contain any false, fictitious or fraudulent statement or entry, shall be fined not more than \$10,000 or imprisoned not more than five years, or both.

September 16, 2003  
Cincinnati, Ohio

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**APPENDIX I**

**HORIBA LA-910****PARTICLE SIZE DISTRIBUTION DATA TABLE Standard 10/25/01**

File Name: Averaged

Sample Name: Averaged Data

ID No:

Dist. Form: SHARP

R.R. Index:

Dist. Mode: NUMBER

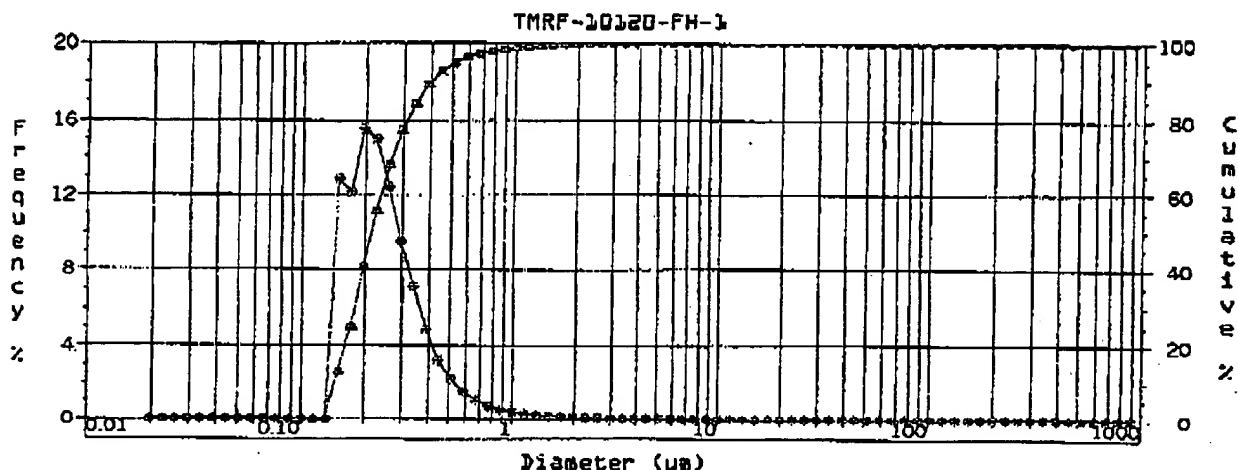
Laser: %

Lamp: %

Circulation: OFF

Agitation: OFF

U.Sonic OFF (min)



No.	SIZE (µm)	FREQ%	UNDR%	No.	SIZE (µm)	FREQ%	UNDR%	No.	SIZE (µm)	FREQ%	UNDR%
(1)	0.020	0.0	0.0	(28)	0.766	0.7	97.9	(55)	29.907	0.0	100.0
(2)	0.022	0.0	0.0	(29)	0.877	0.5	98.4	(56)	34.255	0.0	100.0
(3)	0.026	0.0	0.0	(30)	1.005	0.4	98.8	(57)	39.234	0.0	100.0
(4)	0.029	0.0	0.0	(31)	1.151	0.3	99.0	(58)	44.938	0.0	100.0
(5)	0.034	0.0	0.0	(32)	1.318	0.2	99.2	(59)	51.471	0.0	100.0
(6)	0.039	0.0	0.0	(33)	1.510	0.2	99.4	(60)	58.953	0.0	100.0
(7)	0.044	0.0	0.0	(34)	1.729	0.1	99.5	(61)	67.523	0.0	100.0
(8)	0.051	0.0	0.0	(35)	1.981	0.1	99.6	(62)	77.340	0.0	100.0
(9)	0.058	0.0	0.0	(36)	2.269	0.1	99.7	(63)	88.582	0.0	100.0
(10)	0.067	0.0	0.0	(37)	2.599	0.1	99.8	(64)	101.460	0.0	100.0
(11)	0.076	0.0	0.0	(38)	2.976	0.1	99.8	(65)	116.210	0.0	100.0
(12)	0.087	0.0	0.0	(39)	3.409	0.1	99.9	(66)	133.103	0.0	100.0
(13)	0.100	0.0	0.0	(40)	3.905	0.0	99.9	(67)	152.453	0.0	100.0
(14)	0.115	0.0	0.0	(41)	4.472	0.0	100.0	(68)	174.616	0.0	100.0
(15)	0.131	0.0	0.0	(42)	5.122	0.0	100.0	(69)	200.000	0.0	100.0
(16)	0.150	12.8	12.8	(43)	5.867	0.0	100.0	(70)	229.075	0.0	100.0
(17)	0.172	12.1	24.9	(44)	6.720	0.0	100.0	(71)	262.376	0.0	100.0
(18)	0.197	15.5	40.5	(45)	7.697	0.0	100.0	(72)	300.518	0.0	100.0
(19)	0.226	15.0	55.5	(46)	8.816	0.0	100.0	(73)	344.205	0.0	100.0
(20)	0.259	12.4	67.9	(47)	10.097	0.0	100.0	(74)	394.244	0.0	100.0
(21)	0.296	9.4	77.3	(48)	11.565	0.0	100.0	(75)	451.556	0.0	100.0
(22)	0.339	7.1	84.4	(49)	13.246	0.0	100.0	(76)	517.200	0.0	100.0
(23)	0.389	4.9	89.3	(50)	15.172	0.0	100.0	(77)	592.387	0.0	100.0
(24)	0.445	3.2	92.5	(51)	17.377	0.0	100.0	(78)	678.504	0.0	100.0
(25)	0.510	2.2	94.7	(52)	19.904	0.0	100.0	(79)	777.141	0.0	100.0
(26)	0.584	1.5	96.2	(53)	22.797	0.0	100.0	(80)	890.116	0.0	100.0
(27)	0.669	1.0	97.2	(54)	26.111	0.0	100.0	(81)	1019.510	0.0	100.0

Median : 0.215 (µm) Mean: 0.268 (µm) Mode: 0.184 (µm)  
 Std. Dev.: 0.249 (µm) Span: 31.470  
 Coef. Var: 92.88% Spec. Area: 277711 (cm<sup>2</sup>/cm<sup>3</sup>)

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**APPENDIX II**

**HORIBA LA-910****PARTICLE SIZE DISTRIBUTION DATA TABLE** Standard 10/25/01

File Name: Averaged

Sample Name: Averaged Data

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Dist. F rm: SHARP

R.R. Index:

Dist. Mode: VOLUME

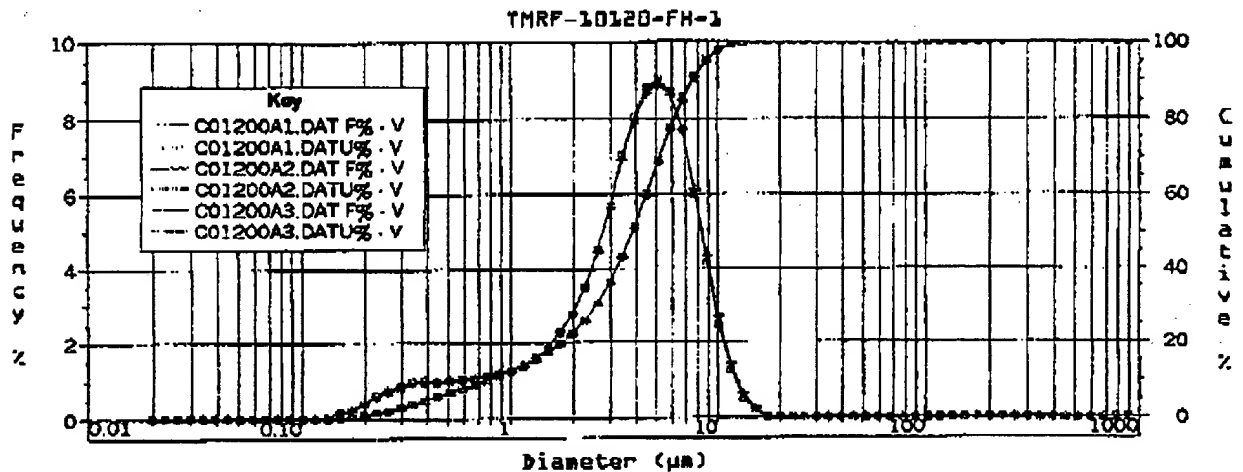
Laser: %

Lamp: %

Circulation: OFF

Agitation: OFF

U.Sonic OFF (min)



No.	SIZE(μm)	FREQ%	UNDR%	No.	SIZE(μm)	FREQ%	UNDR%	No.	SIZE(μm)	FREQ%	UNDR%
(1)	0.020	0.0	0.0	(28)	0.766	1.1	10.1	(55)	29.907	0.0	100.0
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(12)	0.087	0.0	0.0	(39)	3.409	7.0	43.0	(66)	133.103	0.0	100.0
(13)	0.100	0.0	0.0	(40)	3.905	7.9	50.9	(67)	152.453	0.0	100.0
(14)	0.115	0.0	0.0	(41)	4.472	8.7	59.7	(68)	174.616	0.0	100.0
(15)	0.131	0.0	0.0	(42)	5.122	9.0	68.6	(69)	200.000	0.0	100.0
(16)	0.150	0.2	0.2	(43)	5.867	8.7	77.3	(70)	229.075	0.0	100.0
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(27)	0.669	1.1	9.0	(54)	26.111	0.0	100.0	(81)	1019.310	0.0	100.0

Median : 3.946 (μm) Mean: 4.094 (μm) Mode: 4.786 (μm)  
 Std. Dev.: 2.563 (μm) Span: 1.762  
 Coef. Var: 62.66% Spec. Area: 34210 (cm<sup>2</sup>/cm<sup>3</sup>)